Hydrogen Determination in Reactive and Refractory Metals*

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Instrument: RH600/TCH600 (H only application)

Introduction

Titanium is a metal that can be combined with elements such as aluminum and iron to produce high strength, low density, and corrosion resistant alloys. Titanium alloys are used by the military, medical device, sporting good, and aerospace industries because of these properties, and due to the precise nature of these industries, effort needs to be taken to assure that the material meets high quality standards.

One of the most critical chemical specifications of titanium alloys is the hydrogen content. Too high of a hydrogen content can cause hydrides to precipitate, which can lead to embrittlement and subsequent cracking when the alloy is stressed.

The LECO RH600 and TCH600 are hydrogen determinators that utilize an electrode furnace, helium carrier gas, and infrared detection to meet the analytical needs of the ferrous, refractory, and inorganic material industries.

This application note was written for use with the LECO RH600 or TCH600 Hydrogen determinator.

Method Reference

ASTM E1447

Sampling and Sample Preparation

A clean representative sample is required for proper hydrogen determination. Solid samples should be sectioned in such a manner as to avoid overheating, preferably by shearing. If necessary, abrade sample surfaces with a clean file, rinse in acetone, and air dry to remove surface contamination. Cleaned samples must be handled with tweezers or forceps to prevent contamination.

Accessories

782-720 Graphite Crucibles, 761-739 Tin Pellets, 501-059 or 502-040 Tin Capsules, 617-997 Funnel.

Calibration Samples

LECO 502-135, 762-741 Titanium Calibration Samples, NIST or other suitable refractory metal reference materials.

Method Parameters

Analysis Parameters

Outgas Cycles	3
Analysis Delay	20 seconds
Analysis Delay Comparator	1.000
Analysis Type	Semi-Auto Analysis[1]
Auto Analyze on Mass Entry	Disabled
Pre-Analyze Crucible Outgas	Disabled

[1]In earlier software programs this is the same as Auto Analysis. Auto Analysis is now used for instruments equipped with auto-sample loading capability, refer to the latest version of the operator's instruction manual for additional details.

Element Parameters Hydrogen	
Minimum Analysis Time	60 seconds
Significant Digits	6
Conversion Factor	1.000000
Integration Delay	15 seconds
Comparator Level	1.000000%
Stop if below (%)	0.000000

Furnace Parameters	
Furnace Control Mode	Current
Pre-Analyze Purge time	–
Purge Time	10 seconds
Outgas Time	15 seconds
Outgas Cool Time	5 seconds
Outgas Low Power	850 amps*
Outgas High Power	850 amps*
Outgas Ramp Rate	–
Analyze Low Power	800 amps*
Analyze High Power	800 amps *
Analyze Ramp Rate	–
Sample Prep Time	–
Sample Prep Power	–
Temperature Sustain	None

**May vary depending on line voltage. Level can be adjusted to facilitate recovery and/or reduce crucible burn-through.*

Procedure

1. Prepare instrument for operation as outlined in the operator's instruction manual.
2. Determine Blank.
   a. Enter 1.0000 g mass into Sample Login (F3) using Blank as sample name.
   b. Press Loader Switch on front of furnace, after a short delay the loading head slide block will open.
   c. Press Loader Switch again, the loading head slide block will close and the lower electrode will open.
   d. Place two 761-739 tin pellets into a 782-720 graphite crucible.
   e. Place crucible on electrode pedestal.
   f. Press Loader Switch, the lower electrode will close and the analysis sequence will start and end automatically.
   g. Repeat steps 2a through 2f a minimum of three times.
   h. Set the blank following the procedure outlined in the operator's instruction manual.

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3. Calibrate/Drift Correct.
   a. Weigh 0.15 to 0.30 grams of a calibration sample, enter mass, and sample identification into Sample Login (F3).
   b. Press Loader Switch on front of furnace, the loading head slide block will open.
   c. Place sample into open port at top of loading head.
   d. Press Loader switch again, the loading head slide block will close and the lower electrode will open.
   e. Place two 761-739 tin pellets into a 782-720 graphite crucible.
   f. Place crucible on the electrode pedestal.
   g. Press Loader Switch, the lower electrode will close and the analysis sequence will start and end automatically.
   h. Repeat steps 3a through 3g a minimum of three times for each calibration/drift sample used.
   i. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.
4. Analyze Samples.
   a. Weigh 0.15 to 0.30 grams of sample, enter mass, and sample identification into Sample Login (F3).
   b. Proceed as directed in steps 3b through 3g.

Typical Results—Solid Samples

Sample	*Mass g*	*H ppm*
LECO	0.2524	43.0
762-741	0.2540	42.4
Titanium Pin	0.2470	41.6
@42.2 ±3.3	0.2518	42.2
ppm H	0.2484	40.9
	0.2473	42.9
	0.2475	41.1
	0.2515	41.9
	0.2507	40.9
	0.2513	41.5
	***X* =**	***41.9***
	***s* =**	***0.76***

Sample	*Mass g*	*H ppm*
LECO	0.2481	9.9
502-135	0.2478	11.6
Titanium Pin	0.2488	10.3
@11.0 ±2.4	0.2467	10.5
ppm H	0.2494	9.7
	0.2469	12.1
	0.2488	11.8
	0.2471	12.8
	0.2466	11.8
	0.2471	11.4
	***X* =**	***11.2***
	***s* =**	***1.0***

Alternate Procedure—Powder/Chip Samples

If powder or chip samples are to be analyzed, they cannot be placed directly in the loading head. Samples can be weighed into tin capsules and be analyzed in the Semi-Auto Analysis mode. There are issues related to blank, limited volume/sample weight and the increased time and manipulation to weigh samples in a capsule. However, good results and precision are obtainable using this method. In addition, there are two methods of manually loading a sample available to the RH600 user.

Manual Analysis – The electrodes are opened after outgas and the sample is inserted into the crucible. This method will result in higher and more erratic hydrogen blanks and is generally not recommended.

Manual Top Load – After outgas, loading head is opened and the sample is dropped into the crucible. For powder samples, a LECO 617-997 funnel can be inserted through loading head into crucible and the sample is transferred to crucible via the funnel.

This option limits the outgassed crucible exposure to the atmosphere, reducing blank variability, subsequently improving precision.

Procedure—Tin Capsule

1. Determine Blank.
   a. Enter 1.0000 g mass into Sample Login (F3) using Blank as sample name.
   b. Press Loader Switch on front of furnace, after a short delay the loading head slide block will open.
   c. Place an empty tin capsule into open port at top of loading head.

   Note: Use same part number and lot number of capsules that will be used for the analysis of samples, leave capsule open.

   d. Press Loader Switch, the lower electrode will open.
   e. Place two 761-739 tin pellets into a 782-720 graphite crucible.
   f. Place crucible on electrode pedestal.
   g. Press Loader Switch, the lower electrode will close and the analysis sequence will start and end automatically.
   h. Repeat steps 1a through 1g a minimum of three times.
   i. Set the blank following the procedure outlined in the operator's instruction manual.
2. Calibrate/Drift Correct.
   a. Weigh 0.15 to 0.30 grams of calibration sample into a tin capsule, enter mass, and sample identification into Sample Login (F3).

   Note: Leave capsule open.

   b. Press Loader Switch on front of furnace, after a short delay the loading head slide block will open.
   c. Place capsule into open port at top of loading head.
   d. Press Loader Switch, the lower electrode will open.
   e. Place two 761-739 tin pellets into a 782-720 graphite crucible.
   f. Place crucible on electrode pedestal.
   g. Press Loader Switch, the lower electrode will close and the analysis sequence will start and end automatically.

h. Repeat steps 2a through 2g a minimum of three times for each calibration/drift sample used.
i. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.

3. Analyze Samples.
   a. Weigh 0.15 to 0.30 grams of sample into a tin capsule, enter mass, and sample identification into Sample Login (F3).

   Note: Leave capsule open.

   b. Proceed as directed in steps 2b through 2g.

Typical Results—Tin Capsule Method—Powder Samples

Sample	*Mass g*	*H ppm*
Ta Powder	0.2503	195
	0.2573	194
	0.2524	197
	0.2533	194
	0.2522	186
	0.2522	181
	0.2526	190
	0.2551	191
	0.2495	188
	0.2557	198
	***X* =**	***191***
	***s* =**	***5.3***

Procedure—Manual Top Load

1. Set Method Parameters as noted above with the following exceptions.
   a. Under Analysis Parameters set Analysis Type to Manual Top Load.
   b. Under Furnace Parameters set Pre-Analysis Purge Time to 50 seconds.
2. Determine Blank.
   a. Enter 1.0000 g mass into Sample Login (F3) using Blank as sample name.
   b. Press Loader Switch on front of furnace, after a short delay the lower electrode will open.
   c. Place two 761-739 tin pellets into a 782-720 graphite crucible.
   d. Place crucible on electrode pedestal.
   e. Press Loader Switch, the lower electrode will close and the outgas sequence will start automatically.
   f. When the outgas sequence is complete, an add sample message will appear in the lower left hand corner of the instrument display. Press the Loader Switch and the loading head slide block will open.
   g. Place the 617-997 funnel into the open loading head.
   h. Remove the funnel, press the Loader Switch, the loading head slide block will close and the analysis sequence will start and end automatically.
   i. Repeat steps 2a through 2h a minimum of three times.
   j. Set the blank following the procedure outlined in the operator's instruction manual.
3. Calibrate/Drift Correct.
   a. Weigh 0.15 to 0.30 grams of a calibration sample, enter mass, and sample identification into Sample Login (F3).
   b. Press Loader Switch on front of furnace, after a short delay the lower electrode will open.
   c. Place two 761-739 tin pellets into a 782-720 graphite crucible.
   d. Place crucible on electrode pedestal.
   e. Press Loader Switch, the lower electrode will close and the outgas sequence will start automatically.
   f. When the outgas sequence is complete, an "Add Sample" message will appear in the lower left-hand corner of the instrument display. Press the Loader Switch and the loading head slide block will open.
   g. Place the 617-997 funnel into the open loading head and add sample taking care to make sure that all of the sample material is transferred into crucible.
   h. Remove funnel, press Loader Switch; the loading head slide block will close and the analysis sequence will start and end automatically.
   i. Repeat steps 3a through 3h a minimum of three times for each calibration/drift sample used.
   j. Calibrate or Drift Correct the instrument following the procedure outlined in the operator's instruction manual.
4. Analyze Samples.
   a. Weigh 0.15 to 0.30 grams of sample, enter mass, and sample identification into Sample Login (F3).
   b. Proceed as directed in steps 3b through 3h.

Typical Results—Manual Top Load Procedure—Chip Sample

Sample	*Mass g*	*H ppm*
Ti Chip	0.2514	60.5
	0.2524	58.4
	0.2528	55.1
	0.2591	54.4
	0.2583	56.9
	0.2786	59.2
	0.2555	57.4
	0.2581	54.3
	0.2528	59.7
	0.2525	56.5
	***X* =**	***57.2***
	***s* =**	***2.2***

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